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Low Cycle Fatigue Behaviour of a Cu-Cr-Zr-Ti Alloy

A. Biswas^a, A. Nagesha^{b*}, G. Sukumaran^b, P. Parameswaran^b, A.K. Asraff^c,
R. Sandhya^b, S.K. Ray^a

^aDepartment of Materials and Metallurgical Engineering, Jadavpur University, India

^bMetallurgy and Materials Group, Indira Gandhi Centre for Atomic Research, Kalpakkam, India

^cLiquid Propulsion Systems Centre, Valiamala, Thiruvananthapuram, India

Abstract

Cu-Cr-Zr-Ti alloy (Cr: 0.5-0.7, Zr: 0.02-0.05, Ti: 0.02-0.05, Fe: 0.015 and balance Cu) is a candidate material for the inner wall of the double-walled and regeneratively cooled combustion chamber of cryogenic rocket engine. Low cycle fatigue (LCF) behaviour of the alloy was investigated in the solution annealed (1253 K for 10 min. followed by air cooling) condition. Fully reversed, strain controlled LCF tests were performed in air at total strain amplitudes varying from $\pm 0.25\%$ to $\pm 1.0\%$. Different temperatures over 300 to 900 K range were employed for the tests. Additionally, a few tests with fully compressive strain cycling were performed, employing strain amplitudes ranging from -0.5 to -2.0% at a constant temperature of 900 K. All tests were carried out using a fixed strain rate of $3 \times 10^{-3} \text{ s}^{-1}$. The stress response behaviour under fully reversed and mean compressive cycling conditions was observed to be similar for a given total strain range and was seen to be strongly dependent on the temperature of testing. Mean compressive cycling yielded marginally better lives compared to fully reversed cycling. A pronounced initial cyclic hardening followed by a regime of saturation characterised the behaviour at room temperature. However, in the temperature range of 673-773 K, a secondary hardening stage following a saturated stress response was observed. The extent of secondary hardening was seen to be maximum at 673 K. The magnitude of the initial hardening was seen to decrease with increasing temperature. A continuous softening upto failure characterised the stress response behaviour at 900 K. The results are explained in the light of detailed optical and SEM investigations.

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1. Introduction

A rocket engine's combustion chamber is lined with a material that is highly conductive to heat in order to dissipate the huge thermal load. In spite of its high thermal conductivity, pure copper is not a viable candidate for the application due to a low yield strength. A select number of precipitate and dispersion strengthened copper alloys have emerged which possess the requisite balance of good conductivity and good elevated

* Corresponding author:

E-mail address: nagesh@igcar.gov.in

temperature strength. Precipitation strengthened Cu-Ag-Zr alloy, known as NARloy -Z, is one such alloy which is used as a lining material for the main combustion chamber of the space shuttle main engine. In recent times, a similar alloy has been developed as a possible candidate material for the inner wall of a double-walled and regeneratively cooled combustion chamber of the cryogenic rocket engine in the Indian space programme, replacing Ag with less expensive Cr with a minor addition of Ti. Besides, Cu-Cr-Zr alloys are being considered as potential heat sink materials for the plasma facing components of the first wall and divertor of International Thermonuclear Experimental Reactor (ITER) [1-4]. The cyclic mechanical and thermal loads expected in fusion reactor and rocket engine call for a clear understanding of the mechanical properties and in-service microstructural stability of such alloys. The above material differs from the normal commercial variety in that the chemical composition is closely controlled, with lower residual element concentration and a lower inclusion content. The present study addresses the low cycle fatigue and corresponding microstructural evolution of a Cu-Cr-Zr-Ti alloy with the composition (wt. %): Cr: 0.5-0.7, Zr: 0.02-0.05, Ti: 0.02-0.05, Fe: 0.015 and balance Cu.

2. Experimental procedure

The alloy was received in the form of cylindrical blocks of 70 mm diameter and 110 mm length after a solutionising treatment at 1253 K for 10 min followed by air-cooling. The resultant structure was essentially equiaxed, showing a wide range of grain sizes (75-150 μ m) along with a large number of annealing twins (Fig. 1(a)). Blanks of 110mm length and 23 mm diameter were machined along the length direction, using wire EDM.

Fully reversed, strain controlled low cycle fatigue (LCF) tests were performed in air on the alloy at strain amplitudes ($\Delta\epsilon_t/2$) varying from ± 0.25 to $\pm 1.0\%$ at different temperatures in the range, 300 to 900 K. In addition, strain controlled tests with compressive cycling were conducted, keeping the maximum strain at 0 and employing strain amplitudes ranging from -0.5 to -2.0% at a fixed temperature of 900 K. All tests were performed using a constant strain rate of $3 \times 10^{-3} \text{ s}^{-1}$ on an Instron closed-loop servohydraulic test frame. Cylindrical samples of 25mm gauge length and 10mm gauge diameter were used for the tests. Stress-strain hysteresis loops were recorded at intervals throughout the experiments. Fatigue properties were measured from the hysteresis loops obtained at half-life. The fatigue life, N_f was defined as the number of cycles that produced a 20% drop in the saturated tensile load. The failed specimens were longitudinally cut into two pieces across the main cracks situated within the gauge length. One half was used for the optical microscopy and the other, for scanning electron microscopy (SEM) investigations. Samples for optical metallography were swab-etched for 30 seconds using a solution of 15g. Ammonium Persulphate $[(\text{NH}_4)_2\text{S}_2\text{O}_8]$ in 100ml distilled water.

3. Results and discussion

The microstructures of the alloy in the solution-annealed condition and after LCF testing at 900 K are shown in Figs. 1(a) and (b) respectively. Twinning was found to be significant in the alloy as seen in Fig.1 (a), compared to that normally seen in pure copper [1]. Twinning generally gets easy and becomes extensive with a decrease in the stacking fault energy (SFE). The alloy thus appears to be essentially a planar-slip material in contrast to the wavy-slip nature of pure copper. This change in the nature of slip would be expected to pre-empt the common tendency of formation of persistent slip bands (PSBs) observed during the fatigue of pure copper [1, 5]. The chromium present in the alloy does not form intermetallic compounds with copper, rather, it is reported to lead to the formation of Cr-rich precipitates from a supersaturated Cu matrix [6, 7].

The cyclic stress response (CSR) of the material under LCF conditions displayed in general, an initial hardening regime for the first few cycles followed by a well-defined period of saturation which continued till the onset of crack initiation. However, at a temperature of 673 K, the CSR curve showed a pronounced secondary hardening regime, as shown in Figs. 2 and 3. An increase in flow stress, reaching values of 25 to 30%, depending on the imposed strain amplitude was observed in this regime. Upon reaching a peak value, a sharp decline in the response stress was noted. With reduced strain amplitude of $\pm 0.25\%$, the extent of secondary cyclic hardening was seen to be much more pronounced, as can be seen from Fig. 2. This was seen

to be associated with the formation of spherical Cr-rich precipitates as evidenced by the SEM-EDX presented in Figs. 4 (a and b).

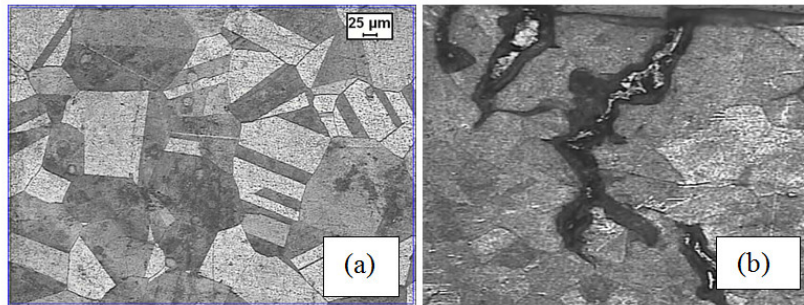


Fig. 1. Microstructure of CuCrZrTi alloy examined by optical microscope: (a) solution-annealed condition, (b) tested at strain amplitude $\pm 1.0\%$ at 900 K.

From Fig. 3 it can also be observed that the life of the material at 673 K is almost identical as that resulted at room temperature, at a strain amplitude of $\pm 0.5\%$. This suggests that the secondary hardening is accompanied by an increase in life. Detailed TEM investigations are necessary in order to establish the operative mechanisms responsible for the occurrence of secondary hardening and understanding the observed life variations. The Variation of number of reversals to failure ($2N_f$) with plastic strain amplitude at different temperatures is presented in Table-1. The generally observed decrease in fatigue life with an increase in the test temperature could be attributed to oxidation as shown in the optical micrograph depicting crack initiation and propagation in the specimen tested at $\pm 1.0\%$ at 900 K (Fig. 1b). Extensive oxide scaling was noticed on the fracture surface of the specimen tested at $\pm 0.5\%$ at 673 K, as shown in Fig. 4(a).

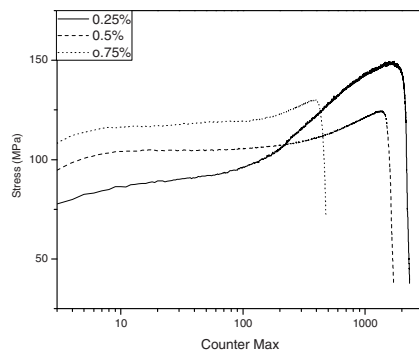


Fig. 2. Cycle stress response at different strain amplitudes (673 K, $3 \times 10^{-3} \text{ s}^{-1}$).

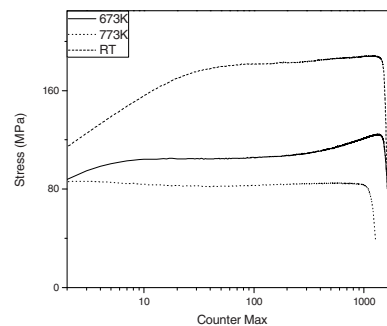


Fig. 3. Cycle stress response at different temperatures ($\pm 0.5\%$, $3 \times 10^{-3} \text{ s}^{-1}$).

Table 1. Strain-life values at different temperatures.

$\Delta\epsilon_t/2$ %	Temperature, K											
	300		573		673		773		873		900	
	$\Delta\epsilon_p/2$ (%)	$2N_f$	$\Delta\epsilon_p/2$ (%)	$2N_f$	$\Delta\epsilon_p/2$ (%)	$2N_f$	$\Delta\epsilon_p/2$ (%)	$2N_f$	$\Delta\epsilon_p/2$ (%)	$2N_f$	$\Delta\epsilon_p/2$ (%)	$2N_f$
± 0.25	-	-	0.071	35482	0.15	4640	0.14	2606	-	-	-	-
± 0.5	0.34	3334	-	-	0.39	3428	0.39	2538	-	-	0.46	2926
± 0.75	0.56	1080	-	-	0.63	958	0.65	920	0.69	1990	0.72	1376
± 1.0	-	-	-	-	0.74	552	-	-	-	-	0.92	278

$\Delta\epsilon_t/2$: total strain amplitude; $\Delta\epsilon_p/2$: plastic strain amplitude

The increase in life with temperature observed under specific testing conditions (Table-1) needs to be probed by further investigations. The alloy also displayed evidence for the occurrence of dynamic strain ageing (DSA) in the temperature range 873-900 K, as reflected by extensive serrations in the stress-strain hysteresis loops as shown in Fig. 5.

Half-life hysteresis loops for fully reversed cycling with a strain amplitude of $\pm 0.5\%$, together with the compressive mean cycling with a strain amplitude of 0 to -1.0% are [presented in Fig. 5. As seen in the figure, fully reversed cycling displayed marginally higher tensile response stress in comparison with that resulted under compressive mean cycling. The variations of cyclic life with total strain for both the fully reversed and compressive mean cycling conditions are presented in Fig. 6. It can be noted that the former yielded slightly lower lives, which is consistent with the variation in the tensile stress response that has a strong bearing on the cyclic life.

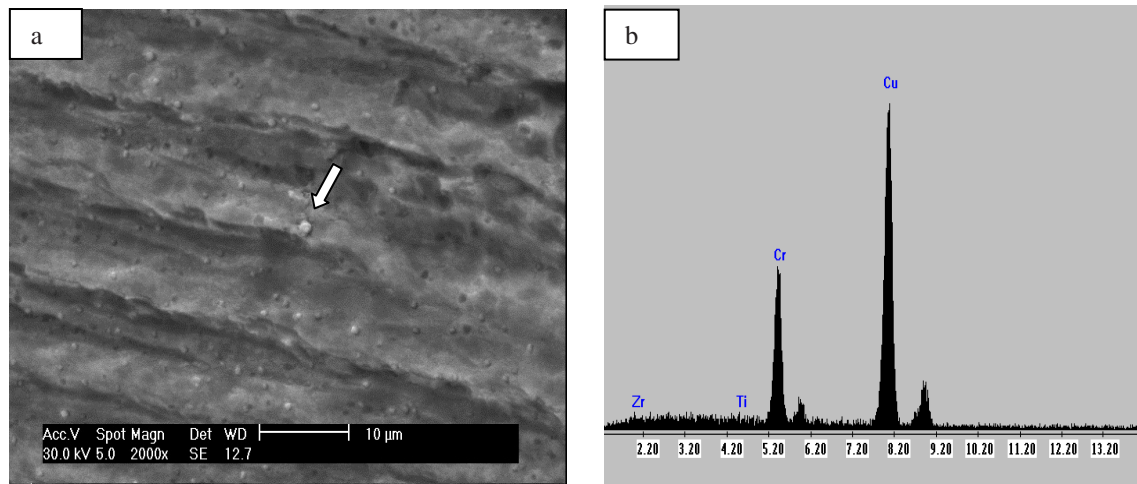


Fig. 4 (a): SEM fractograph pertaining to LCF test at 673 K with a strain amplitude of $\pm 0.25\%$, (b): EDX pattern obtained from the precipitate indicated with arrow.

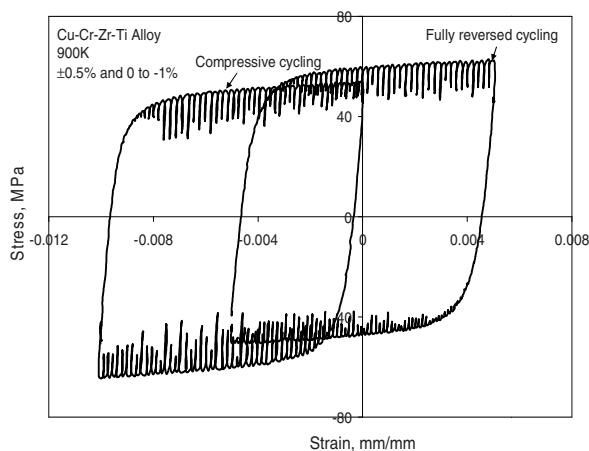


Fig. 5. Half-life hysteresis loops for fully reversed and compressive cycling (strain rate: $3 \times 10^{-3} \text{ s}^{-1}$, temperature: 900 K).

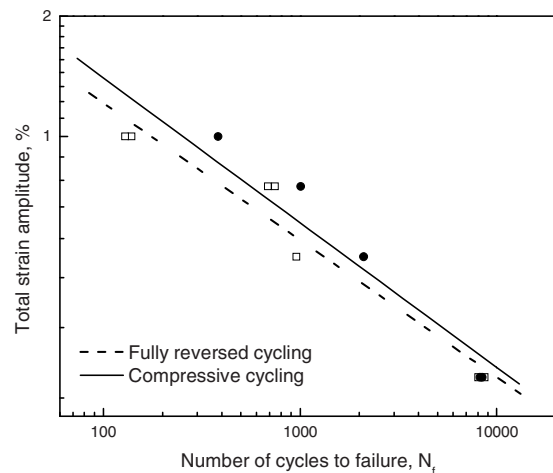


Fig. 6. Total strain vs. cycle to failure plot for fully reversed and compressive mean cycling (strain rate: $3 \times 10^{-3} \text{ s}^{-1}$, temperature: 900 K).

4. Conclusions

- The cyclic stress response of the alloy showed a brief initial hardening followed by saturation. A pronounced secondary hardening, associated with the formation of spherical Cr-rich precipitates was observed under LCF cycling in the temperature range, 673-773 K.
- The alloy displayed dynamic strain ageing in the temperature range of 873 to 900 K.
- Compressive mean stress yielded a marginally better life compared to an equivalent fully reversed cycling.

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